

## Analysis of a Tributyl Borane/Methoxypropylamine sample by Gas Chromatography Mass Spectrometry

INTRODUCTION

One (1) tributyl borane/methoxypropylamine (TBB/MOPA) sample, labeled as H-29, was submitted by Mark Sonnenschein for the quantitative determination of tributyl borane in water by mass spectrometry. Accurately measure aliquots of the TBB/MOPA sample were diluted (10% v/v) into three solvents: (1) acetonitrile, (2) acetonitrile/water mixture (1/1 v/v) and (3) water. Aliquots of these three solutions were analyzed immediately after preparation and approximately 16 hours later for comparison by gas chromatography mass spectrometry (GC/MS) operating in the electron impact (EI) ionization mode.

EXPERIMENTAL

Accurately measured aliquots (0.1 milliliters) of the TBB/MOPA sample were diluted into one (1) milliliter aliquots of (1) acetonitrile, (2) water/acetonitrile mixture (1/1 v/v) and (3) water. One (1) microliter aliquots of these TBB/MOPA solutions were analyzed immediately after preparation and approximately 16 hours later on a Finnigan SSQ7000, SN TS010023, quadrupole GC/MS system operating in the electron impact (EI) ionization mode. Representative analytical conditions were as follows:

Instrument internet name - mdassq7k  
Instrument model - SSQ 7000  
Instrument serial number - TS010023  
Workstation internet name - mdssqd2  
ICIS Version 8.1.1  
DEC OSF/1 V2.0 (Rev. 240); Thu May 19 08:21:51 PDT 1994  
DEC OSF/1 V2.0 Worksystem Software (Rev. 240)  
File name: 3082pk6  
Sample: TBB-MOPA/H2O/ACN 50/50 TIME=0  
Operator: pk/SSQ7000  
Comments: EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5 50/1  
Study: MD-2000-003082  
Client: M. Sonnenschein  
Injected volume: 1.0  
Analysis started at 18-APR-00 16:10:08 Analysis will stop when GC program stops  
Tune file name: cal041100.ict from 11 APR 0 5:44  
DSP Version 2.7  
GC descriptor: msonn-2 ICL procedure: essex20  
Vacuum status -> ok  
Manifold temperature 70.301 C Manifold pressure 3.267 e-7 Collision cell pressure 0  
Ion source type = Electron ionization  
Scan mode = Q1MS 2584 u mass range / Positive ions  
Full scan -> First mass = 49.971 amu Last mass = 59.989 amu  
Scan time = 1 seconds Scan rate = 10 amu/seconds  
Ionization Mode: EI IONIZATION Requested Source Temp: 150 deg Actual Source Temp: 146 deg  
GC descriptor msonn-2  
Injector: 300 deg 330 deg max Transfer Line: 320 deg 350 deg max  
Column: 60 deg at 0.0 minutes 340 deg max  
60 deg at 2.0 minutes 320 deg at 22.0 minutes  
Valve B: open Valve A: open Stabilize time 0.1 minutes  
At retention time 0 Min. -> Filament is off Electron multiplier = 0 V Electrometer zero = 0  
Analysis stopped at 18-APR-00 16:40:48 (retention time 29.95 Min) Analysis stopped because gc run finished

The TBB/MOPA in acetonitrile/water and water only solutions were briefly mixed on a vortex mixer prior to analysis to insure homogeneity. After sitting idle for more than 10 minutes these solutions were noted to partition into two (2) phases.

RESULTS

Representative computer reconstructed total ion chromatograms plus tentative assignments from the GC/MS/EI analyses of TBB/MOPA in acetonitrile, water/acetonitrile and water only solutions at Time = 0 hours are presented in Figures 1 thru 3, respectively. The area counts of the MOPA peak (identified as Amine on Figures 1 thru 3) were used as an internal standard to ratio the response of the TBB peaks in the three solutions. Assuming no TBB decomposition takes place in the TBB/MOPA in acetonitrile solution

Ratio TBB in ACN / TBB in H2O = 22.3 to 2.15 10 fold decrease in TBB concentration at time = 0  
for 1/1 water/acetonitrile solution 22.3 to 3.34 = 7 fold decrease at time = 0

Similar results for 16 hr samples, no further decrease in TBB concentration,  
Need more water? Rate determining step or due to partition of organic/water layer observed

Solution ID	Area Counts Amine	Area Counts TBB peaks	Area Counts TBB Bioxin	Ratio Area Counts TBB/Amine	Ratio Area Counts TBB Bioxin/Amine
TBB/MOPA in Acetonitrile Time = 0 hrs	9.02E+08	2.01E+10	1.185E+09	22.3	1.31
TBB/MOPA in Water/Acetonitrile Time = 0 hrs	1.11E+09	3.712E+09	4.01E+08	3.34	0.36
TBB/MOPA in Water Time = 0 hrs	9.34E+08	2.01E+09	4.01E+08	2.15	0.43
TBB/MOPA in Acetonitrile Time = 16 hrs	1.66E+09	2.335E+10	1.798E+09	14.07	1.08
TBB/MOPA in Water/Acetonitrile Time = 16 hrs	1.84E+09	7.816E+09	6.99E+08	4.25	3.80
TBB/MOPA in Water Time = 16 hrs	1.40E+09	2.915E+09	1.84E+09	2.08	1.31

Mass spectra and raw data are filed with this report.

REFERENCES

1. P. E. Kastl, Raw Data Envelope, MD 2000-003082.

FIGURE 1

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Acetonitrile solution at Time = 0 hours.

CHRO: ms7k3082pk4 18-APR-00 Elapse: 11:50 1350  
Samp: TBB-MOPA/ACN 10% TIME=0 Start: 14:51:53 2942  
Comm: EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5 100/1  
Mode: EI +Q1MS LMR UP LR Study: MD-2000-003082  
Oper: pk/SSQ7000 Client: M. Sonnenschein Inlet: GC  
Peak: 1000.00 mmu Label wndw: 200 > 1500 Masses: 20 > 400  
Area: 0, 4.00, 0 Baseline: 0, 3 Label: 0, 40.0

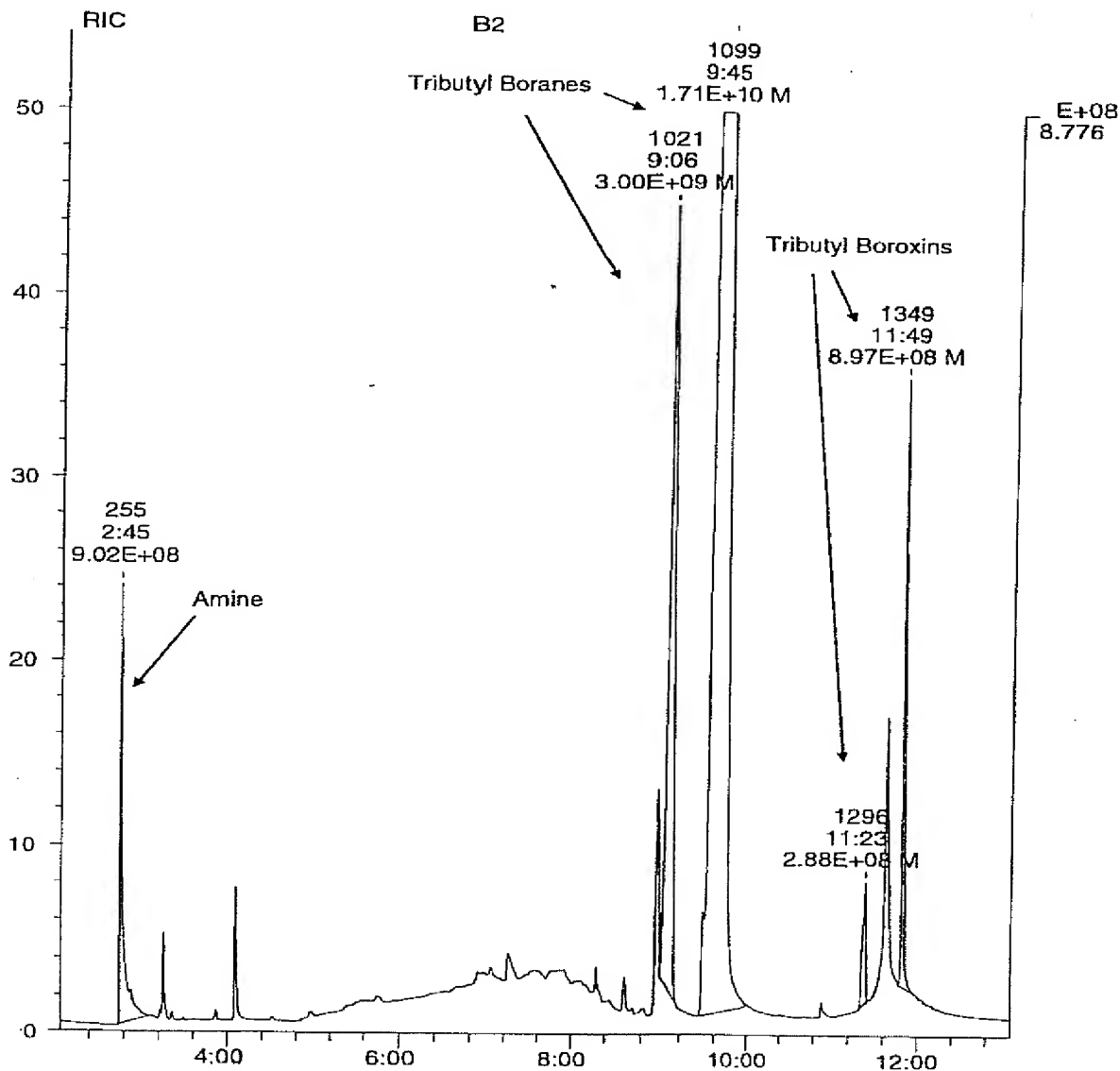


FIGURE 2

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Water/Acetonitrile solution at Time = 0 hours.

CHRO: ms7k3082pk6  
Samp: TBB-MOPA/H2O/ACN 50/50 TIME=0  
Comm: EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5 50/1  
Mode: EI +Q1MS LMR UP LR  
Oper: pk/SSQ7000 Client: M. Sonnenschein  
Peak: 1000.00 mmu Label wndw: 200 > 1500  
Area: 0, 4.00, 0 Baseline : 0, 3

18-APR-00 Elapse: 11:40 1396  
Start : 16:10:08 3276  
Study : MD-2000-003082  
Inlet : GC  
Masses: 20 > 400  
Label : 0, 40.0

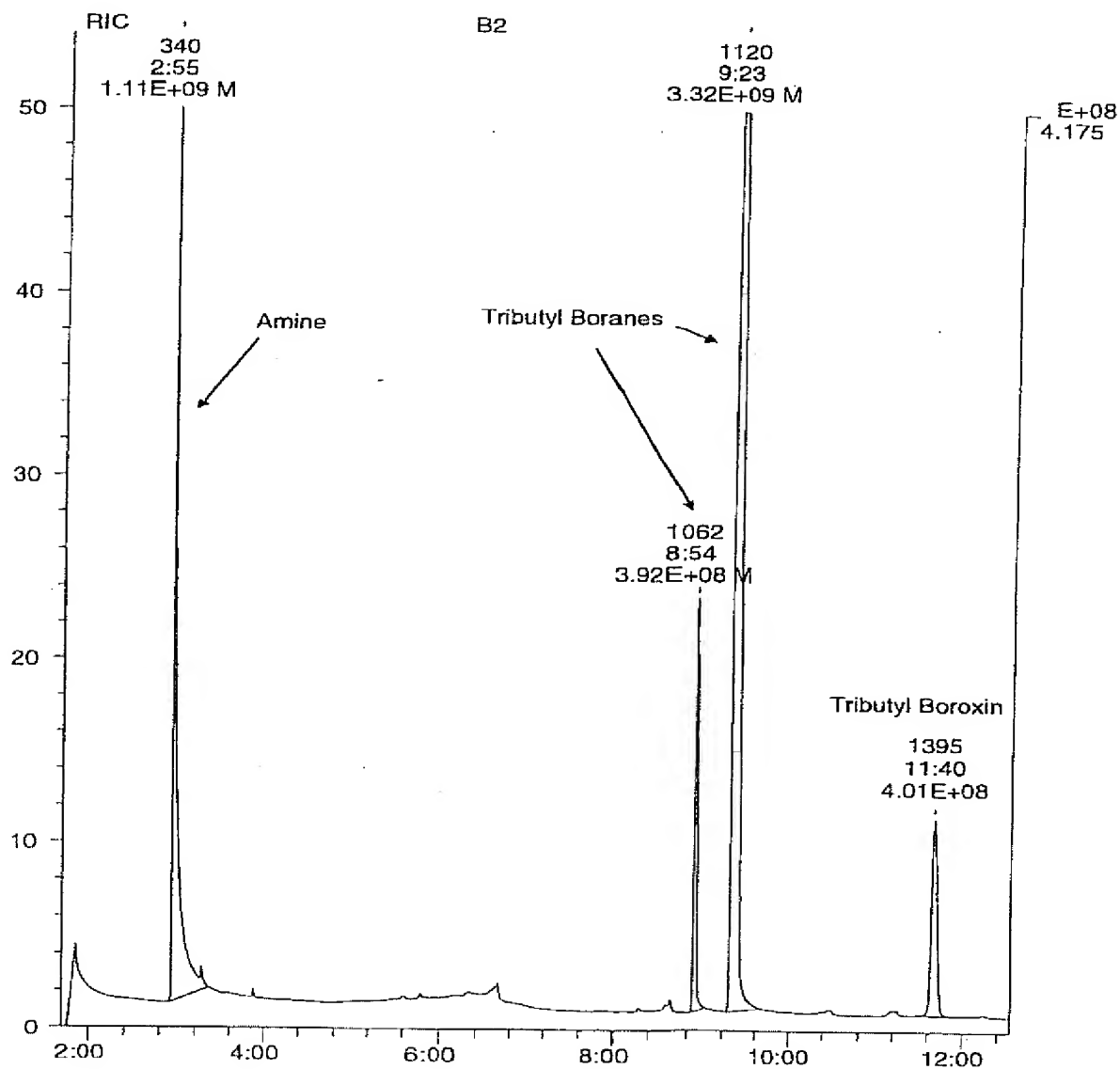


FIGURE 4

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Acetonitrile solution after approximately 16 hours.

CHRO:	ms7k3082pk7	19-APR-00	Elapse:	11:42	1396
Samp:	TBB-MOPA/ACN 10% TIME=12+ hours		Start :	06:01:44	3274
Comm:	EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5		50/1		
Mode:	EI+Q1MS LMR UP LR		Study :	MD-2000-003082	
Oper:	pk/SSQ7000	Client: M. Sonnenschein	Inlet :	GC	
Peak:	1000.00 mmu	Label wndw: 200 > 1500	Masses:	20 > 400	
Area:	0, 4.00, 0	Baseline : 0, 3	Label :	0, 40.0	

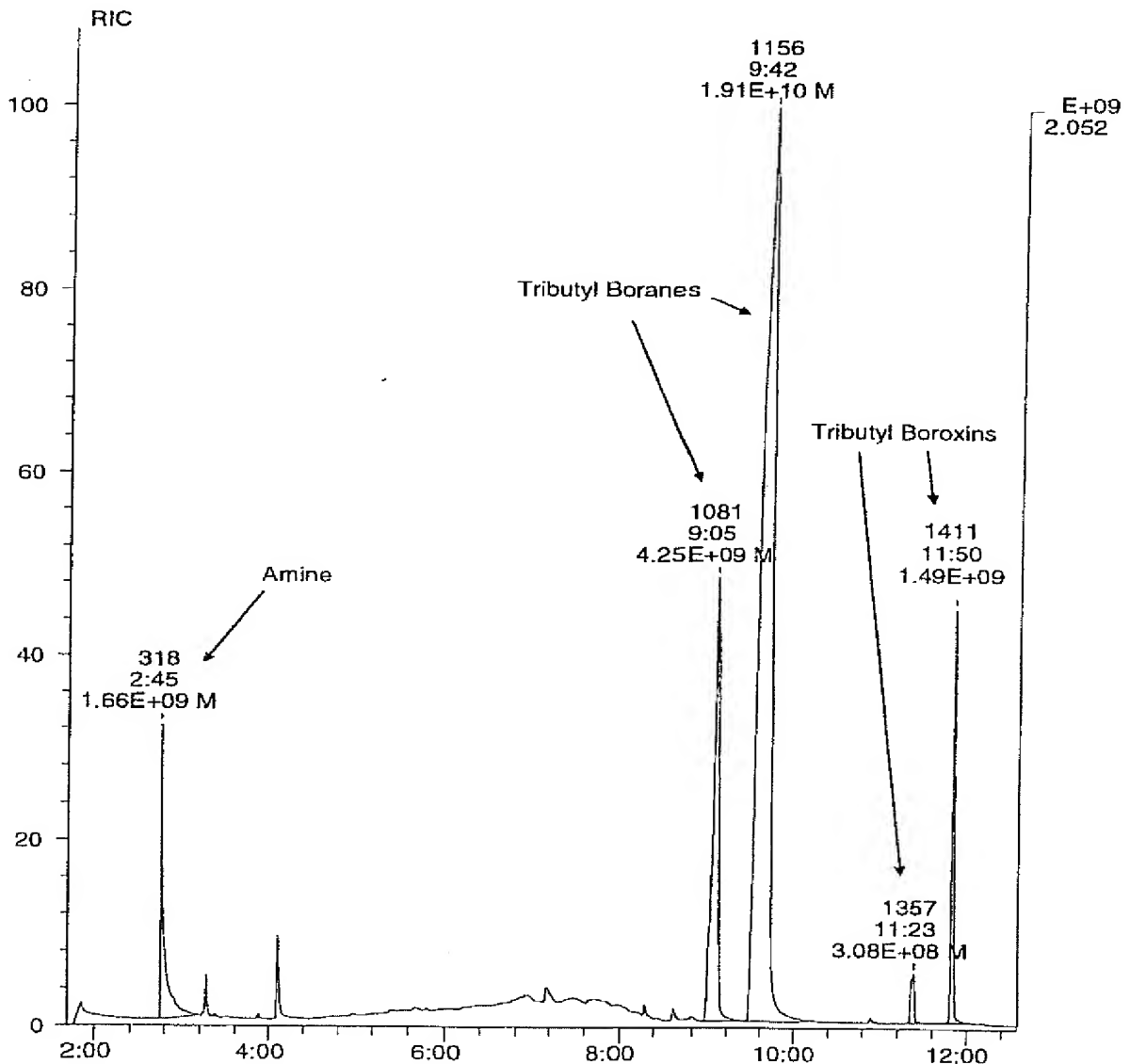


FIGURE 3

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Water (10% v/v) at Time = 0 hours.

CHRO:	ms7k3082pk5	18-APR-00	Elapse:	11:42	1396
Samp:	TBB-MOPA/H2O 10% TIME=0		Start:	15:27:45	3273
Comm:	EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5	100/1			
Mode:	EI +Q1MS LMR UP LR		Study:	MD-2000-003082	
Oper:	pk/SSQ7000	Client: M. Sonnenschein	Inlet:	GC	
Peak:	1000.00 mmu	Label wndw: 200 > 1500	Masses:	20 > 400	
Area:	0, 4.00, 0	Baseline: 0, 3	Label:	0, 40.0	

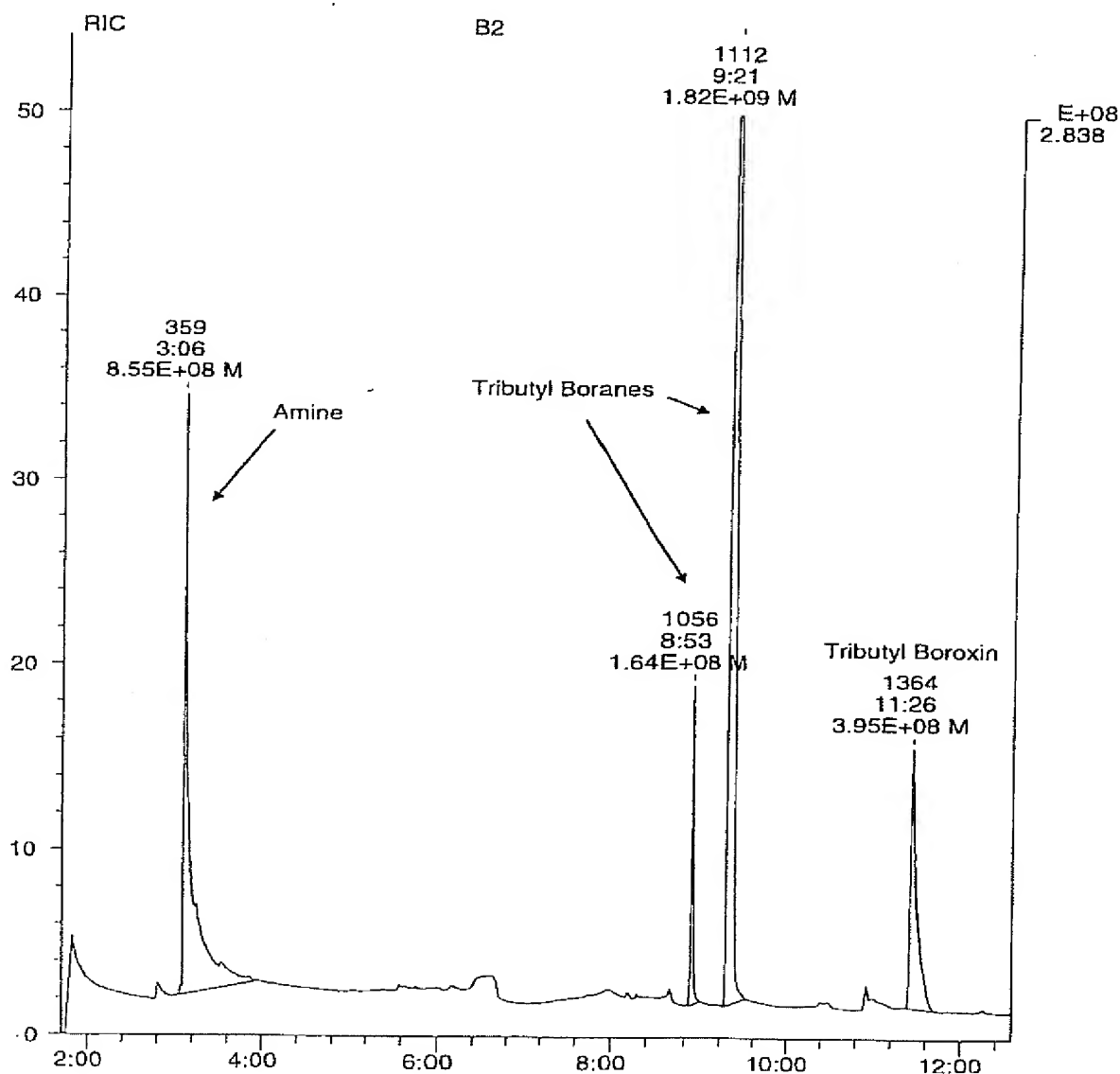


FIGURE 5

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Water/Acetonitrile solution after approximately 16 hours.

CHRO:	ms7k3082pk8	19-APR-00	Elapse:	06:01.4	712
Samp:	TBB-MOPA 10% in 50/50 ACN/H2O TIME=12+		Start :	08:12:07	3274
Comm:	EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5	50/1			
Mode:	EI +Q1MS LMR UP LR		Study :	MD-2000-003082	
Oper:	pk/SSQ7000	Client: M. Sonnenschein	Inlet :	GC	
Peak:	1000.00 mmu	Label wndw: 200 > 1500	Masses:	20 > 400	
Area:	0, 4.00, 0	Baseline : 0, 3	Label :	0, 40.0	

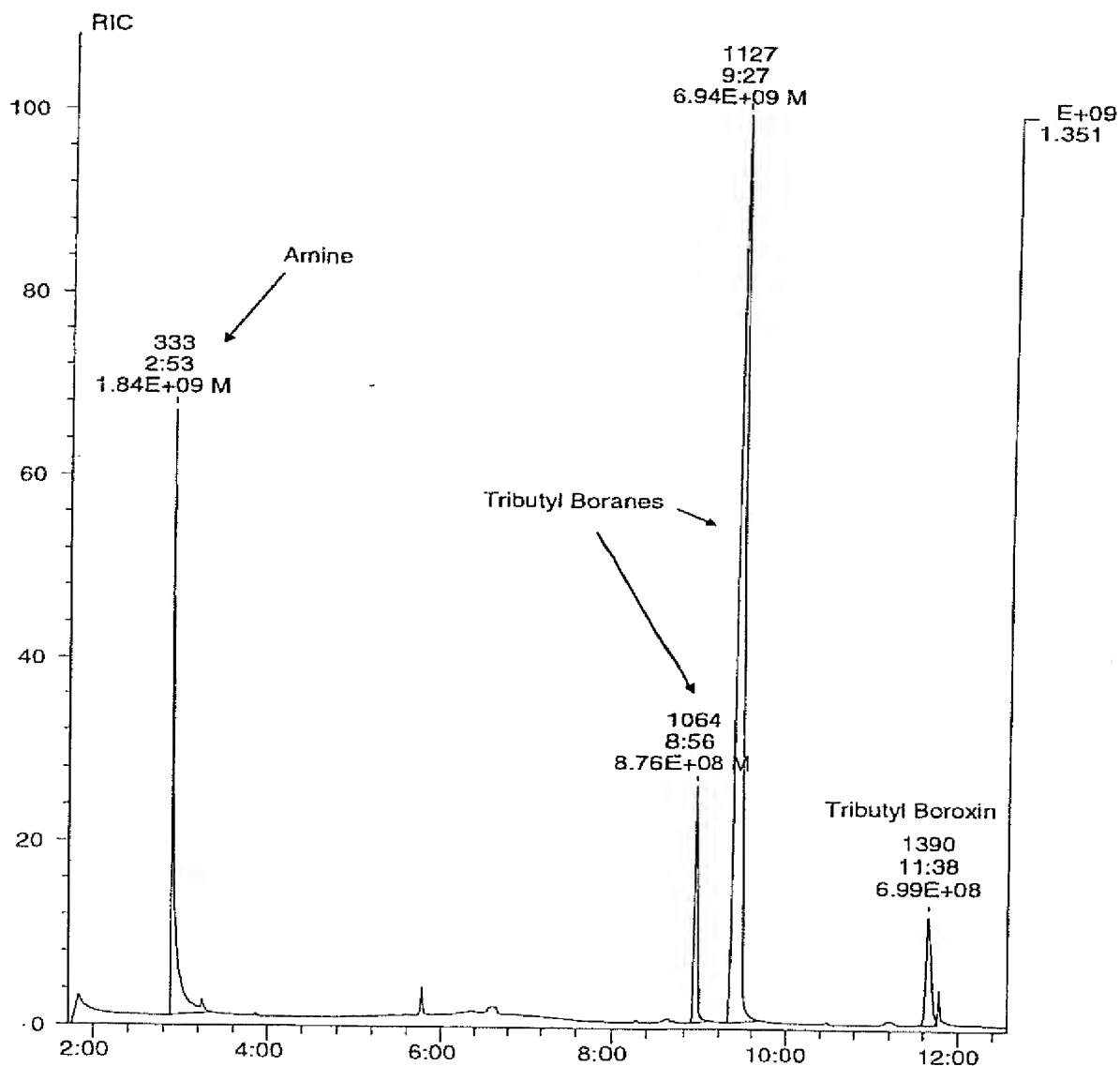


FIGURE 6

Representative computer reconstructed total ion chromatogram plus tentative assignments from the GC/MS/EI analysis of a TBB/MOPA in Water solution after approximately 16 hours.

CHRO:	ms7k3082pk9	19-APR-00	Elapse:	11:34	1382
Samp:	TBB-MOPA IN 10% v/vH2O 2ml TIME=12+		Start :	08:53:07	3274
Comm:	EI/GC/MS 20x0.18x.4u Rtx-5 60(2)-13-320 m/z 20-400@0.5		50/1		
Mode:	EI +Q1MS LMR UP LR		Study :	MD-2000-003082	
Oper:	pk/SSQ7000	Client: M. Sonnenschein	Inlet :	GC	
Peak:	1000.00 mmu	Label wndw: 200 > 1500	Masses:	20 > 400	
Area:	0, 4.00, 0	Baseline : 0, 3	Label :	0, 40.0	

